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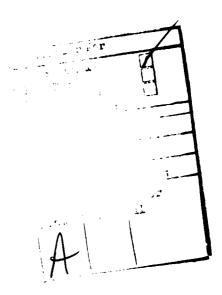
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### **PREFACE**

The investigation reported herein was authorized by a first indorsement dated 8 May 1974 from the Office, Chief of Engineers, U. S. Army, to a U. S. Army Engineer Waterways Experiment Station (WES) letter dated 29 April 1974, subject: Project Plan for Investigation of Testing Methods and Apparatus, Tests of Proprietary Admixtures (CWR Work Unit 31138). The Technical Monitor for this investigation was Mr. J. A. Rhodes, DAEN-CWE-C.

The investigation was conducted during 1977 and 1978 at the Structures Laboratory, WES, under the supervision of Messrs. B. Mather, Acting Chief of the Structures Laboratory, and J. M. Scanlon, Jr., Chief of the Engineering Mechanics Division. The members of the staff actively concerned with the work included Messrs. K. L. Saucier and W. B. Lee. Mr. Saucier prepared this report.

Commanders and Directors of WES during this investigation and the preparation of this report were COL John L. Cannon, CE, and COL Nelson P. Conover, CE. Technical Director was Mr. F. R. Brown.



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# CONVERSION FACTORS, CUSTOMARY INCH-POUND TO METRIC (SI) UNITS OF MEASUREMENT

Customary inch-pound units of measurement used in this report can be converted to metric (SI) units as follows:

Multiply	By	To Obtain
inches	25.4	millimetres
cubic yards	0.7645549	cubic metres
pounds (mass)	0.4535924	kilograms
pounds (mass) per cubic yard	0.59327631	kilograms per cubic metre
pounds (force) per square inch	0.006894757	megapascals

### INVESTIGATION OF PROPRIETARY ADMIXTURES

1977-1978 Tests

PART I: INTRODUCTION

### Background

1. Subsequent to completion of the work described in Report 1,\* the Structures Laboratory received word of products which were said to be of a nature that would allow a proper air-void system to be obtained and retained in air-entrained concrete. Samples of three such admixtures were obtained by the laboratory and were tested for resistance to freezing and thawing, since all of the previously tested admixtures (as described in Report 1) generally failed to comply with this requirement.

### Purpose

2. This investigation was conducted to evaluate certain water- reducing admixtures for use in concrete in Corps of Engineers Civil Works construction.

### Scope

3. Two admixtures (designated SM-10-77 and SM-1-78) were evaluated according to the applicable provisions of CRD-C 87-78\*\* (ASTM C 494-77) for their acceptability as type A, water-reducing admixtures. One admixture (SM-5-79) was evaluated for compliance with the requirements of type D, water-reducing and set-retarding admixture. Tests were conducted

<sup>\*</sup> W. O. Tynes. 1977. "Investigation of Proprietary Admixtures," Technical Report C-77-1, U. S. Army Engineer Waterways Experiment Station, CE, Vicksburg, Miss.

<sup>\*\*</sup> U. S. Army Engineer Waterways Experiment Station, CE. 1949 (with quarterly supplements). Handbook for Concrete and Cement, Vicksburg, Miss.

for determination of water content, time of setting, compressive strength, and resistance to freezing and thawing.

## Revisions and Corrections to Report 1

- 4. Since Report 1 was published, errors have been found in the report and should be corrected as follows:
  - $\underline{\mathbf{a}}$ . Delete last two sentences of paragraph 31, p 21.
  - <u>b</u>. In Table 5, column headed "Relative DFE at 300 Cycles," delete "Relative."
  - c. In Table 5, last column, change "in." to "%."
  - d. In Table 6 change as follows: Delete all length change data; substitute the following:

Test Length change, %:	Mixture A B Batch No. (1,2,3) (1,2,3)	Mixture A C Batch No. (4,5,6) (1,2,3)	Mixture A D Batch No. (7) (1)	Mixture A E Batch No. (8) (1)	Mixture A C1 Batch No. Speci- (9) (1) fied
Increase over control	-0.002	-0.001			+0.010 max
	Mixture	Minh	M2	W.:	Milandana
	A B1 Batch No.	Mixture	Mixture	Mixture	Mixture
		A E1	A C2	A B2	A B2(2)
	(10,11)	Batch No.	Batch No.	Batch No.	Batch No. Speci-
Tanakh	(1,2)	(12) $(1)$	(12) $(1)$	<u>(13)</u> (1)	(13) (1) <u>fied</u>
Length change, %:					
Increase over					
control	-0.002	-0.004	-0.005	+0.004	-0.009 +0.010 max

### PART II: MATERIALS, MIXTURES, AND TESTS

### Materials

### Portland cement

5. Type II portland cement (RC-705) from Alabama was used in this investigation. Table 1 presents the results of chemical and physical tests of the cement.

### **Aggregates**

6. The natural fine aggregate (WES S-4(51)) and the crushed limestone coarse aggregate (CL-2 G-1(3)) were obtained from Mississippi and Tennessee, respectively. The aggregates were graded to meet the requirements of CRD-C 87-78 (ASTM C 494-7). Table 2 gives the gradings and results of physical tests of the aggregates.

### Air-entraining admixture

7. The air-entraining admixture (AEA-937) used in this investigation was a solution of neutralized vinsol resin.

### Chemical admixture

- 8. The three commercially available proprietary admixtures obtained for this investigation were described as follows:
  - a. SM-10-77 is a condensation product of melamine and formaldehyde.
  - b. SM-1-78 is a sulfonated naphthalene formaldehyde condensate.
  - c. SM-5-79 is an aqueous solution of a modified naphthalene sulfonate.

### Mixtures

9. Air-entrained concrete mixtures, containing 25.0-mm (1-in.)\* nominal maximum size aggregate, were proportioned with and without the chemical admixtures under test. The cement content of all mixtures was 517 lb/yd $^3$  (307 kg/m $^3$ ); slump was 2-1/2  $\pm$  1/2 in. (63  $\pm$  13 mm) for all

<sup>\*</sup> A table of factors for converting customary inch-pound units of measurement to metric (SI) units is presented on page 3.

mixtures; and the air content when determined by CRD-C 41 (ASTM C 231), the pressure method, was  $6.0 \pm 0.5$  percent for all mixtures. The difference between the air content of the control concrete and that of the concrete containing the admixture under test did not exceed 0.5 percent. Pertinent information on the mixtures is given below:

Mixture	Chemical Designation	Admixture Amount Used*	Water- Cement Ratio, by Weight	No. of Batches
A	None		0.45	9
F	SM-10-77	3.0	0.37	3
G	SM-1-78	0.8	0.37	2
G1	SM-1-78	0.7	0.40	1
н	SM-5-79	1.7	0.36	3

<sup>\*</sup> Percent of solution by weight of cement.

Mixtures A through E were used in the basic investigation (Report 1).

- 10. All concrete mixtures were proportioned, batched, mixed, sampled, and cured according to the requirements of CRD-C 87-78 with three exceptions: (1) A reference laboratory Type II cement was used rather than a blend as specified in CRD-C 87-78. (2) The quantity of air-entraining admixture varied with the different admixtures and also with the amount of the admixtures used. (3) Since the manufacturers recommended a mixing procedure different from that specified, the recommendations of the manufacturers were followed. They were:
  - a. For admixtures SM-10-77 and SM-5-79:
    - (1) Place coarse aggregate in the mixer.
    - (2) Put air-entraining admixture (AEA) in mixing water.
    - (3) Put half of mixing water in mixer, mix two turns.
    - (4) Add fine aggregate and cement, mix two turns.
    - (5) Add remainder of mixing water, mix 1/2 min.
    - (6) Add water-reducing admixture.
    - (7) Mix 3 min., rest 3 min., remix 2 min.

- b. For admixture SM-1-78:
  - (1) Place coarse aggregate in the mixer.
  - (2) Add half of mixing water, mix two turns.
  - (3) Add fine aggregate and cement, mix two turns.
  - (4) Add AEA and water-reducing admixture.
  - (5) Add remainder of mixing water.
  - (6) Mix 3 min., rest 3 min., remix 2 min.

### Tests

11. Three batches of test concrete and three control batches (mixture A) were made for each admixture. One slump, air content, and time-of-setting test were made on each batch cast. Three 6- by 12-in. (152- by 305-mm) cylinders from each batch were tested for compressive strength at ages of 3 days, 7 days, 28 days, 6 months, and 1 yr. Two beams from mixture H and three beams from all other mixtures were tested for resistance to freezing and thawing according to procedure A of CRD-C 20-77 (ASTM C 666-77). Mixture data and results of tests of freeshly mixed concrete are given in Table 3.

### PART III: TEST RESULTS

### Water Reduction

12. The amount of water reduction obtained with high-range water reducers is generally proportional to the amount of admixture used, up to a practical limit. The manufacturers of the three admixtures tested herein advised that reductions in the range of 15 to 20 percent should be used in order to secure the proper air-void system for frost resistance. Therefore, the water reductions were selected to meet this criterion. Table 3 indicates that all of the admixtures far exceeded the 5 percent reduction required by CRD-C 87 (ASTM C 494). A determination of the practical limit of water reduction for the products was not made.

### Time of Setting

13. Time of setting was determined by ASTM Method C 403. Results of tests are given in Table 4. Mixtures F (SM-10-77) and H (SM-5-77) met the requirements for type A water-reducing admixture (ASTM C 494). Mixture G (SM-1-78) met the requirements for type D, water-reducing and retarding admixture.

### Compressive Strength

14. The results of the compressive strength tests, given in Table 4, indicate the strength gain to be commensurate with the water reductions obtained. The results, also plotted in Figure 1, greatly exceed the test requirements. The strength gain, determined as a percent of the control, is on the order of 200 percent at early ages and 120 to 140 percent for long-term strength.

### Resistance to Accelerated Freezing and Thawing

15. Results of tests of specimens subjected to accelerated

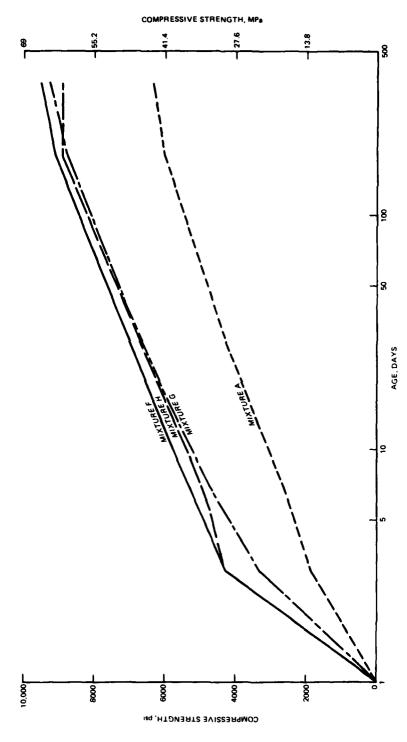


Figure 1. Average compressive strength for control mixture (A) and three test mixtures

freezing and thawing tests are given in Table 5. There was some concern that the method of consolidation might have an effect on the air-void system and hence on frost resistance. Consequently, two of the test beams from each batch of mixture F were consolidated by rodding and one by internal vibration. Indications are that the method of consolidation has no effect on the results of the test for resistance to freezing and thawing. A relative durability factor of 104 percent was obtained on mixture F compared with the control; therefore, no significant difference is indicated between the frost resistance of the concrete made with and without the high-range water reducer.

- 16. Three beams were consolidated by rodding and tested for each round (batch) of mixture G. However, the frost resistance of mixture G which had a water reduction of 17 percent did not meet the requirements of the specification. A relative durablilty factor of only 60 was obtained for two test rounds. The dosage of admixture SM-1-78 was reduced so as to obtain a 12 percent water reduction (mixture G1). At this dosage the admixture gave a relative durability factor of 98, and thus met the requirements for frost resistance for the one round tested. Although no tests were conducted at intermediate dosage rates, it may be postulated that a dosage to secure approximately 15 percent water reduction might permit the necessary void system to exist to meet the relative durability factor requirement of 80 minimum.
- 17. Air-void spacing tests, conducted on three test specimens of mixtures G and G1, support the findings of the tests for resistance to freezing and thawing. A spacing factor  $(\overline{L})$  of 0.0033 in. (0.083 mm) was obtained on a control specimen with good frost resistance, and a factor of 0.0082 in. (0.208 mm) was obtained on a test specimen with poor resistance. A maximum of 0.008 in. (0.20 mm) is taken as the critical value for frost resistant concrete. The specimen from mixture G1 had good resistance and an  $(\overline{L})$  of 0.0067 in. (0.170 mm).
- 18. The results of test on mixture H are not conclusive as those on mixtures F and G. Mixture H had the greatest water reduction of the three admixtures tested, i.e. 20 percent. Two beams were tested for resistance to freezing and thawing from each of three test rounds

(batches) and three control rounds. The relative durability factor was 78 which is only two less than the requirement. A lower admixture dosage could possibly have resulted in satisfactory frost resistant concrete in terms of the specification if admixture SM-5-78 acts in a manner similar to SM-1-78.

### PART IV: DISCUSSION

- 19. The results of this study demonstrate that frost resistant, air-entrained concrete can be made using some high-range water-reducing admixtures. Whether such concrete can be obtained consistently under field conditions has not yet been demonstrated on Corps projects.
- 20. Of particular significance is the apparent difference in degree of frost resistance obtained with different dosages of the admixtures. Clearly, all high-range water-reducing admixtures contemplated for use in environments where concrete may freeze while critically saturated should be checked for frost resistance capability using job materials, mixtures, and dosage rates. Based on the work of this study and the previous investigation, indications are that high-range water-reducing admixtures would meet all other requirements of CRD-C 87 (ASTM C 494).
- 21. The results reported herein indicate that durable, high-quality concrete can be obtained with high-range water-reducing admixtures when certain conditions are met. At the International Symposium on Superplasticizers (sic), the benefits and drawbacks were fully discussed.\* In order to realize the full potential of and minimize the problems associated with the use of high-rage water-reducing admixtures, several areas would appear to require further study, including:
  - a. Slump loss shortly after mixing.
  - b. Mixture proportioning, particularly with regard to the grading and amount of fine aggregate.
  - c. Interaction chemically with various cements.
  - d. Amount and time of dosage.
  - e. Interaction with other admixtures, including airentraining agents.
  - f. Finishability.

<sup>\*</sup> Superplasticizers in Concrete, American Concrete Institute SP-62, 1979, 427 pp.

Table 1

Chemical and Physical Properties

of Portland Cement

Properties	RC-705
Chemical Data	
SiO <sub>2</sub> , percent	22.8
Al <sub>2</sub> O <sub>3</sub> , percent	4.0
Fe <sub>2</sub> 0 <sub>3</sub> , percent	4.2
MgO, percent	3.5
SO <sub>3</sub> , percent	1.7
Loss on ignition, percent	0.6
Insoluble residue, percent	0.26
Na <sub>2</sub> 0, percent	0.12
K <sub>2</sub> O, percent	0.49
Total alkalies, Na <sub>2</sub> O, percent	0.44
C <sub>3</sub> S, percent	45.6
C <sub>3</sub> A, percent	3.5
C <sub>2</sub> S, percent	30.9
CaO, percent	62.8
C <sub>4</sub> AF, percent	12.7
Physical Data	
Specific gravity	3.15
Fineness, air permeability, cm <sup>2</sup> /g	3150
Time of set, Gillmore:	
Initial, hr:min	3:15
Final, hr:min	5:45
Mortar expansion, autoclave test, percent	0.10
Air content, percent	8.4
Compressive strength, psi (MPa)	
3 days	1630 (11.24)
7 days	2280 (15.72)

Table 2

Gradings and Physical Properties

of Fine and Coarse Aggregates

	Coarse Aggregate CL-2 G-1(3)	Fine Aggregate (Sand) WES-1 S-4(51)
Grading	s, Percent Passing	
Sieve Size:		
25.9-mm (1-in.)	100	~ •
19.0-mm (3/4-in.)	75	
12.5-mm (1/2-in.)	50	~~
9.5-mm (3/8-in.)	25	
4.75-mm (No. 4)		100
2.36-mm (No. 8)		88
1.18-mm (No. 16)		73
600-μm (No. 30)		47
300-μm (No. 50)		16
150-μm (No. 100)		3
75-μm (No. 200)	~-	
Passing 75-μm (No. 200)		
Phy	sical Properties	
Absorption, percent	0.4	0.2
Specific gravity	2.72	2.64
Fineness modulus	7.00	2.73

Reduction Percent Water 18 18 18 18 Chemical Admixture 15.51 15.51 15.51 15.51 4.14 4.14

3.00 3.00 3.00

SM-10-77

Avg

15 16 14

3.00

Avg

| | |

AEA 3 m1/yd3

Percent Content

5.8 5.8 5.8

57.2 63.5 63.5

2-1/4 2-1/2 2-1/2 2-1/2

0.45 0.45 0.45

in.

by Weight

Water-Cement Ratio

Percent by Weight of Cement

Designation

Batch No.

Mixture

Mixture Data and Results of Freshly Mixed Concrete

Table 3

178

9.0

63.5

0.45

620 620 620

5.8 6.5 6.0

57.2 57.2 63.5 57.2

2-1/4 2-1/4 2-1/2

0.37 0.37 0.37

620

6.1

2-1/4

0.37

178 178

5.6 6.3

57.2 63.5

2-1/4 2-1/4

0.45

178

6.0

63.5

2-1/2

0.45

85 85

5.6 5.8

69.8 63.5

2-3/4 2-1/2

0.37

0.80

SM-1-78

G

Avg

17 18

0.80

Avg

63.5

2-1/2

0.37

85

178 100

63.5 63.5

2-1/2 2-1/2

12

3.62

0.70

SM-1-78

A G1

Made a second of

4

(Continued)

Table 3 (Concluded)

			Chemical Admixture	dmixture						
			Percent		Water	Water- Cement			Air	
	Batch		by Weight		Reduction	Ratio	Slı	dun	Content	AEA 3
Mixture	No.	Designation	of Cement		Percent	by Weight	in.		Percent	m1/yd
¥	20	;	;		:	0.45	2-1/5	63.5	5.8	125
	21	:	;		;	0.45	2-1/2	63.5	6.1	125
	22	:	;		:	0.45	2-1/5	63.5	5.8	125
	Avg	;	;	;	i I	0.45	2-1/2 63.5	63.5	5.9	125
×		SM-5-79	1.70	8.84	20	0.36	7	50.8	5.8	130
	7		1.70	8.84	20	0.36	2-1/5	63.5	6.5	130
	ო		1.70	8.84	20	0.36	2-1/2	63.5	6.3	130
	Avg		1.70	8.84	20	0.36	2-1/4	57.2	6.2	130

Table 4

Results of Tests of Setting Time and Compressive Strength

	Batch		ime of tial		nal			Compress	ive Stren	eth. nsi	
Mixture	No.	hr	min	hr	min		3 d	7 d	28 d	6 mo	1 y
	14	5	45	7	40		1890	2710	4680	6540	6890
^	15	5	50	8	0		1730	2540	4340	6180	6480
	16	5	55	7	55		1960	2750	4360		
			_								
	Avg	5	50	7	50		1860	2760	4460	6360	6680
F	1	5	1.5	7	1.0		4040	5410	7320	9360	9546
	2	5	35	7	0		4160	5410	6870	8860	9520
	3	5	35	7	5		4520	5360	6890		
	Avg	5	30	7	5		4240	5390	7030	9110	9530
Diff from C	ontrol:		-20		-45	۲:	228	202	158	143	143
Required, T	ype A:* -1	hr to +	1-1/2 h	r		%:	110	110	110	100	100
A	17	5	5	7	25		1950	2590	4210	6250	6430
	18	5	25	7	35		2000	2730	4360	6480	6650
	Avg	5	15	7	30		1980	2660	4280	6360	6540
G	1	8	35	10	25		2680	4360	6660	8620	889
	2						4020	5250	6910	8960	9590
	Avg	8	35	10	25		3350	4800	6780	8790	9240
Diff from C	ontrol:	+3	30	+2	55	%:	169	180	158	138	141
Required, T	ype D:* +1	hr to +	3-1/3 h	r		%:	110	110	110	100	100
A	19	5	30	7	35		1730	2500	3890	5680	6190
G1	1	7	25	9	20		3410	4390	5930	7920	8570
Diff from C	ontrol:	+1	55	+1	45	%:	197	176	152	139	138
A	20	5	45	8	0		1880	2520	4040	5660	6290
	21	5	20	7	35		1700	2320	3840	5480	5360
	22	5	50	8	7		1960	2700	4250	6020	5610
	Avg	5	38	7	55		1850	2510	4040	5720	5750
н	1	6	0	7	55		4430	5230	7040	9250	8540
	2	5	45	7	35		4090	5080	6890	8890	9070
	3	7	0	9	7		4130	4860	6250	8640	9070
	Avg	6	15	8	26		4220	5060	6730	8930	8890
Diff from C			+37		+31	%:	228	202	167	156	155
Required T	ype A:* -1	hr to t	1-1/2 h	r		%:	110	110	110	100	100

<sup>\*</sup> By CRD-C 87 (ASTM C 494).

Table 5 Results of Tests for Relative Durability Factor (RDF) and Bubble-Spacing Factor (1)

	Batch	Du	rability F	actor		Bubble- Spacing Factor
Mixture	No.	Rodded	Rodded	Vibrated	RDF	$(\overline{L})$ , in.
Α	14	82	86	87		
Α	15	62 45*	71	73		
	16	78	63	77		
	Avg		77		104	
F	1	86	85	80		
	2	79	78	71		
	3	84	75	79		
	Avg		80			
		Rodded	Rodded	Rodded		
Α	17	80	79	74		
	18	83	79**	79		0.0033**
	Avg		79		60	
G	1	41**	45	43		0.0082**
	2	61	54	42		
	Avg		48			
A	19	77	80	71		
	Avg		76		98	
<b>G</b> 1	1	75	77**	71		0.0067**
	Avg		74			
Α	20	83	86			
	21	83	84			
	22	79	76			
	Avg		82		78	
Н	1	54	55			
	2	71	71			
	3	66	65			
	Avg		64			

 $<sup>^{\</sup>star}$  Deleted from average. \*\*  $\overline{L}$  determined on specimen indicated by \*\* after DFE value.

In accordance with letter from DAEN-RDC, DAEN-ASI dated 22 July 1977, Subject: Facsimile Catalog Cards for Laboratory Technical Publications, a facsimile catalog card in Library of Congress MARC format is reproduced below.

Saucier, Kenneth Lamar

Investigation of proprietary admixtures; Report 2: 1977-1978 tests / by Kenneth L. Saucier. Vicksburg, Miss.: U. S. Waterways Experiment Station; Springfield, Va.: available from National Technical Information Service, 1980. 13, [6] p.: ill.; 27 cm. (Technical report - U. S. Army Engineer Waterways Experiment Station; C-77-1, Report 2) Prepared for Office, Chief of Engineers, U. S. Army, Washington, D. C., under CWR Work Unit 31138.

1. Admixtures. 2. Concrete admixtures. 3. Water reducing agents. I. United States. Army. Corps of Engineers. II. Series: United States. Waterways Experiment Station, Vicksburg, Miss. Technical report: C-77-1, Report 2. TA7.W34 no.C-77-1 Report 2

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